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<b>(21) International Application Number:</b> PCT/US00/10177 <b>(22) International Filing Date:</b> 14 April 2000 (14.04.00)  <b>(30) Priority Data:</b> 09/291,697      14 April 1999 (14.04.99)      US 09/383,456      26 August 1999 (26.08.99)      US  <b>(71) Applicant:</b> CHARVID LIMITED LIABILITY COMPANY [US/US]; 6731 E. 50th Avenue, Commerce City, CO 80022 (US).  <b>(72) Inventor:</b> TALLEY, Charles, Bullick; 2235 Mountain Sage Drive, Highlands Ranch, CO 80126 (US).  <b>(74) Agents:</b> SWARTZ, Douglas, W. et al.; Sheridan Ross P.C., Suite 1200, 1560 Broadway, Denver, CO 80202-5141 (US).		<b>(81) Designated States:</b> AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).  <b>Published</b> <i>With international search report.</i>
<b>(54) Title:</b> METHOD AND COMPOSITION FOR CLEANING BEVERAGE LINES  <b>(57) Abstract</b> <p>The present invention provides a method and cleaning compositions for removing oxalate, protein, and/or microbial deposits from food preparation, packaging, and dispensing devices. The method first contacts and acid cleaner with the device, second contacts an alkaline cleaner with the device, and third contacts an antimicrobial composition with the device.</p> <div data-bbox="1071 1134 1461 1470" data-label="Image"> </div>		

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## METHOD AND COMPOSITION FOR CLEANING BEVERAGE LINES

### FIELD OF THE INVENTION

5 The method and composition of the present invention is generally directed to cleaning and sanitizing food and beverage equipment and specifically to cleaning and sanitizing beverage dispensing equipment.

### BACKGROUND OF THE INVENTION

10 In beverage dispensing equipment, particularly in beverage dispensing lines, various types of deposits can form that not only provide off-flavors and off-odors to beverages that run through the lines but also create health hazards. These deposits are generally known as mineral or oxalate deposits, protein deposits, and microbial deposits (e.g., bacteria, yeast, and mold). Because it is often impractical and costly to replace the dispensing lines, the lines are cleaned periodically in an attempt to remove the foreign  
15 deposits.

Existing line cleaning procedures run caustic cleaners through the lines at temperatures ranging from about 36 to about 40 °F for typical cleaning times of only 5 to 10 minutes to attempt to remove these deposits. Longer cleaning times cause unnecessary and costly interruptions in business, especially in the bar and restaurant  
20 industries.

These procedures suffer from a number of problems. First, the cleaners are not selective for both mineral and protein deposits and therefore fail to realize a high degree of removal of both types of foreign deposits. Second, at cleaning temperatures of 36 to 40°F the sodium hydroxide in the caustic cleaner will begin to crystallize with the crystals  
25 being too big to remove foreign deposits imbedded in the polymer structure of the cleaning line. Third, caustic cleaners are highly corrosive to certain objects, such as brass pumps and taps. Fourth, the caustic cleaner has only limited sanitizing efficacy, thereby failing to remove adequately the microbial deposits. Finally, caustic cleaners are typically difficult to rinse at low temperature and require extensive line flushing with the beverage  
30 before a dispensing line can be reused. The need for extensive flushing consumes unnecessary amounts of the beverage.

## SUMMARY OF THE INVENTION

It is an objective of the present invention to provide an effective cleaning composition and methodology for removing mineral or oxalate deposits, protein deposits, and microbial deposits from beverage dispensing equipment. Related objectives include providing a cleaning composition and methodology that can quickly clean beverage dispensing equipment, that is effective at temperatures of 36°F or higher, that is noncorrosive to metal objects, such as brass pumps and taps, and that has a highly efficacious in removing microbial deposits.

In one embodiment of the present invention, a method for cleaning and sanitizing not only beverage dispensing lines but also other types of food processing, preparation, packaging and dispensing equipment is provided. The method includes the steps of:

(a) contacting an article having at least two of a mineral deposit, a protein deposit, and a microbial deposit with an acidic cleaning composition to at least partially remove any mineral or oxalate deposits;

(b) contacting the article with an alkaline cleaning composition to at least partially remove any protein or other organic deposits; and

(c) contacting the article with an antimicrobial composition to at least partially remove any microbial deposits. The method provides an effective cleaning composition and methodology for removing mineral or oxalate deposits, protein deposits, and microbial deposits from beverage dispensing equipment. The method can also quickly clean beverage dispensing equipment, can be effective at temperatures of 36°F or higher, can be noncorrosive to metal objects, such as brass pumps and taps, and can be highly efficacious in removing microbial deposits.

The acidic cleaning composition can be any acid cleaner and preferably includes at least one of nitric acid, sulfuric acid, hydrochloric acid, and mixtures thereof to remove the mineral and/or protein deposits. Another acid, such as phosphoric acid, oxalic acid, citric acid, hydroxyacetic acid and mixtures thereof, is used to stabilize the previously noted acid. More preferably, the acidic cleaning composition includes nitric acid and phosphoric acid. Nitric acid is able to remove both oxalate and protein deposits. The acid cleaning composition can include a surfactant to facilitate removal of the oxalate deposits. Phosphoric acid inhibits the surfactant from reacting with the nitric acid. The acid cleaner

is effective at low temperatures (i.e., of about 40°F and less) and typically does not generate heat when removing unwanted deposits, which is highly advantageous on beverage dispensing applications. The alkaline cleaning composition can be any suitable alkaline cleaner. For example, the alkaline cleaner can be a caustic alkaline cleaner such as that described in copending U.S. Application Serial No. 08/874,086, filed on February 25, 1999 and copending U.S. Application entitled "Cleaning Composition Having Low Impurities" Serial No. 09/291,696, filed on April 14, 1999 or a non-caustic alkaline cleaner such as that described in U.S. Patents 5,898,024, 5,863,345, 5,789,361, and 5,663,132, and copending U.S. Application Serial Nos. 09/299,376, filed April 27, 1999, 09/128,060, filed August 3, 1998 and 09/129,060, filed August 4, 1998, all of which are incorporated herein by reference in their entireties. More preferably, the alkaline cleaner includes a hydroxide, a peroxygen compound, and/or mixtures thereof as the primary cleaning agent. Even more preferably, the alkaline cleaning composition includes: (a) from about 25 to about 40 wt% of a peroxygen compound; (b) from about 15 to about 40 wt% of a silicate; and (c) from about 2 to about 8 wt% of a chelate; or includes: (a) at least about 35 wt% of a hydroxide; (b) from about 2 to about 5 wt% of an organic salt derived from the reaction of gluconic acid with the hydroxide; (c) from about 0.1 to about 3 wt% of a gluconate; and (d) from about 1 to about 8 wt% of an inorganic compound.

The antimicrobial composition can include any suitable antimicrobial agents. A preferred antimicrobial composition includes: (a) a solvent; (b) no more than about 14 wt% of a sulfonated compound; (c) from about 0.001 to about 8 wt% of a solubilizing agent; and (d) an antimicrobial agent. This composition is described in detail in copending U.S. Application Serial No. 09/023,347, filed February 13, 1998, which is incorporated herein by reference in its entirety.

In another embodiment of the present invention, the process for cleaning and sanitizing beverage dispensing devices includes the following steps:

(a) contacting a beverage dispensing device having at least one of a mineral deposit, a protein deposit, and a microbial deposit with an acidic cleaning composition to at least partially remove any mineral deposits and

(b) contacting the device with an alkaline cleaning composition to at least partially remove any protein deposits.

In yet another embodiment of the present invention, an aqueous acid cleaner is provided that includes:

(a) at least one of nitric acid, sulfuric acid, and hydrochloric acid;  
(b) at least one of phosphoric acid, oxalic acid, citric acid, and hydroxyacetic acid; and

(c) a surfactant. The cleaner preferably includes from about 15 to about 40 wt% and more preferably from about 15 to about 25 wt% nitric acid; from about 8 to about 30 wt% and more preferably from about 10 to about 15 wt% of phosphoric acid; and from about 1 to about 4 wt% surfactant. The surfactant is preferably selected from the group consisting of a nonphenol, polyoxyethylene-polyoxypropylene glycols, octyl phenols, phosphate esters, ethylene oxides, and mixtures thereof.

In yet another embodiment, an acidic cleaning composition is provided. The composition includes an aggressive acid that is at least one of nitric acid, sulfuric acid, and hydrochloric acid and a stabilizing acid that is at least one of phosphoric acid, oxalic acid, citric acid, and hydroxyacetic acid to stabilize the aggressive acid.

#### BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a cross-sectional view of a beverage dispensing line showing the portion of the foreign deposit that is commonly removed by conventional cleaning methodologies.

Figs. 2A and 2B are respectively a cross-sectional view and an enlarged view of a beverage dispensing line showing the typical penetration of foreign deposits into the polymeric structure of the line.

Fig. 3 is a flow schematic depicting a method according to the present invention.

#### DETAILED DESCRIPTION

The method and cleaning compositions of the present invention overcome the shortcomings of conventional beverage dispensing device cleaning methodologies and compositions. As used herein, a beverage dispensing device includes not only taps, tap lines, and storage vessels but any other device used in the dispensing of beverages. As noted above, the method and cleaning compositions of the present invention are not

limited to beverage dispensing devices but are equally applicable to food processing, preparation, packaging, and dispensing equipment.

The method and cleaning composition of the present invention was developed at least in part based on the recognition of the reasons underlying the shortcomings of conventional caustic cleaners. By way of example, Figure 1 depicts a cross-sectional view of a beverage dispensing line after cleaning using conventional caustic cleaners. While not wishing to be bound by any theory, it is believed that the deposit coating the interior of the line 14 is partially removed at the bottom of the line 14 and relatively untouched in other portions of the line 14. This unequal removal is caused by the inability of typical caustic cleaners to selectively remove both the oxalate and protein deposits that form in the line 14. Because the velocity of the caustic cleaner is greatest at the bottom of the line 14, most of the deposits that are removed by the cleaner are located at the bottom of the line 14. When a carbonated beverage, such as beer, is run through the line after cleaning, the effervescence of the beverage removes additional portions of the deposits causing off-odors or off-flavors in the dispensed beverage. While again not wishing to be bound by any theory, it is believed that the caustic cleaner's ability to remove the deposits is further limited by the depth of penetration of the deposit into the porous surface of the line 14. Figures 2A and 2B depict the porosity of the line 14 and the penetration of the deposits into the porous surface. Referring to Figure 2B, the porosity of the line 14 due to molecular spacing between polymers is depicted on a microscopic scale. As shown in Figure 2A, oxalate, protein and/or microbial deposits penetrate into the intermolecular spaces between the polymers. Because conventional caustic cleaners are typically used in a line 14 at or near the freezing point of the caustic component (e.g., 36 to 40°F), the caustic forms large crystals which are unable to penetrate into the spaces between the polymers to remove the deposits. Finally, carbon dioxide is commonly used to push the caustic cleaner through the line. The carbon dioxide reacts with the hydroxide to form carbonic acid. The carbonic acid decreases the pH of the caustic cleaner, thereby reducing significantly the efficacy of the caustic in removing the deposit.

Referring to Figure 3, a methodology is presented that overcomes each of these restrictions on the cleaning of beverage dispensing devices.

In an optional first step 20, an acid cleaner is contacted with the beverage dispensing device to remove oxalate deposits and weaken the bonds between the protein and/or microbial deposits and the surface of the device. While not wishing to be bound by any theory, it is believed that the acid in the acid cleaner reacts with alkaline metals in the oxalate deposits to weaken and thereby remove the deposits. Oxalate deposits commonly are comprised of calcium oxalate. Oxalate deposits typically form by the reaction between salts and proteins in a beverage, particularly fermented beverages, which causes a product of the reaction to precipitate out of the beverage and form oxalate deposits. The acid cleaner can be any acid cleaner having a pH less than about pH 4 and containing one or more aggressive acids that readily react with the oxalate deposits.

The preferred acid cleaner is a liquid mixture of various acids. More preferably, the acid cleaner includes an aggressive acid such as nitric acid, sulfuric acid, hydrochloric acid, and mixtures thereof with nitric acid being even more preferred. The concentration of the aggressive acid preferably ranges from about 2.5 to about 6 mole/l and more preferably from about 5.5 to about 4.5 mole/l. Additionally, the acid cleaner preferably includes a second acid different from the first acid to stabilize the aggressive acids, which can be highly volatile in solution. Preferred stabilizing acids include phosphoric acid, oxalic acid, citric acid and mixtures thereof, with phosphoric acid being more preferred. The preferred concentration of the stabilizing acid preferably ranges from about 1 to about 3 mole/l and more preferably from about 1.4 to about 2.1 mole/l. Preferably, the molar ratios between the aggressive acid and the stabilizing acid ranges from about 2:1 to about 3:1 and more preferably from about 2.5:1 to about 2.8:1.

The acid cleaner can further include a surfactant (or wetting agent) which acts as an emulsifier to facilitate removal of oxalate and protein deposits. Preferred surfactants include nonphenols, octylphenols, polyoxyethylene-polyoxypropylene glycols, phosphate esters, ethylene oxides and mixtures thereof with polyoxyethylene-polyoxypropylene glycols and nonphenols being more preferred. The acid cleaner preferably includes from about 0.006 to about 0.03 moles/l and more preferably from about 0.012 to about 0.02 moles/l of the surfactant. The molar ratio between the aggressive acid and the surfactant preferably ranges from about 350:1 to about 100:1 and more preferably from about 308:1 to about 154:1.



The remainder of the acid cleaner is preferably water. Typically, the acid cleaner includes from about 60 to about 80 wt% water and has a pH ranging from about pH 0.1 to about pH 1.

5 The method to prepare the acid cleaner is relatively straightforward. Water is placed in a suitable noncorrosive mixing vessel and agitated. Next, the stabilizing acid is added to the water. After mixing is completed, the aggressive acid is next added to the mixture. Again after mixing is completed, the surfactant is added. The mixture is then agitated for a time preferably ranging from about 15 to about 30 minutes and more preferably from about 20 to about 25 minutes. Finally, to provide coloring of the acid  
10 cleaner 1 gram of a coloring agent such as methylene blue is added for every 1,000 pounds of cleaner.

As noted above, the step 20 is optional and is commonly used to remove off-odors and off-flavors and oxalate deposits from beverage dispensing equipment. In step 20, the acid cleaner is preferably diluted with water at a volumetric ratio of about 150 to about  
15 30 and more preferably from about 70 to about 40 parts water to one part acid cleaner. The diluted acid cleaner is introduced into the beverage dispensing device and preferably left in contact with the device for a residence time of at least about 5 minutes and more preferably ranging from about 8 to about 12 minutes. As will be appreciated, the diluted acid cleaner can be introduced into the beverage dispensing device under pressure to  
20 displace any beverage remaining in the device.

The aggressive acid is small enough in size to penetrate into the spaces between polymers in the device and remove at least a portion of the embedded oxalate and, to a lesser extent, protein deposits.

Referring again to Figure 3, an alkaline cleaner is next contacted 24 with the  
25 beverage dispensing device primarily to remove remaining oxalate and protein deposits. The alkaline cleaner can be a caustic cleaner or a non-caustic cleaner. Typically, the alkaline cleaner has a pH ranging from about pH 10 to about pH 13.5.

A preferred caustic cleaner is a liquid and has a low freezing temperature which preferably is less than about 32°F. Preferred caustic cleaners include (a) at least about  
30 35% by weight of a hydroxide (with sodium hydroxide being more preferred); (b) from about 2 to about 5 wt% of an organic salt derived from the reaction of gluconic acid with

the hydroxide; (c) from about 0.1 to about 3 wt% of a heptagluconate; and (d) from about 1 to about 8 wt% of an inorganic compound such as a potassium hydroxide. The cleaner preferably includes no more than about 750 ppm carbonates, no more than about 19 ppm iron, no more than about 19 ppm chlorites, no more than about 77 ppm sulfates, no more than about 0.4 ppm mercury, and no more than about 750 ppm chlorides. The remaining constituent of the cleaner is commonly water.

The caustic cleaner is preferably made by first reacting the hydroxide with gluconic acid in a vessel originally containing the hydroxide, second contacting the mixture with the inorganic compound, third contacting the mixture of the second step with water, and finally contacting the mixture of the third step with a gluconate.

The other preferred alkaline cleaner is non-caustic and is described in detail in U.S. Patents 5,863,345; 5,789,361; and 5,663,132. The non-caustic alkaline cleaner is preferably in the form of a free-flowing particulate mixture that includes (a) from about 25 to about 35 wt% of a peroxygen compound (such as a coated peroxygen compound sold by Burlington Chemical under the tradename "FB"®); (b) from about 25 to about 35 wt% of a metasilicate and/or sesquisilicate; (c) from about 4 to about 6 wt% and more preferably about 5 wt% of a chelate selected from the group consisting of EDTA, NTA, and other derivatives of a carboxylic acid and phosphoric acid and derivatives thereof and mixtures thereof; (d) from about 25 to about 45 wt% of a builder selected from the group consisting of a sulfate, a phosphate, a carbonate and mixtures thereof; (e) from about 2.5 to about 5 wt% of a surfactant such as a primary alkane sulfonate sold under the trademark BIO TERG PAS-8S, propylene oxide and ethylene oxide block polymer sold under the trademark PLURONIC L-61, nonylphenoxypoly (ethyleneoxy) ethanol sold under the trademark IGEPAL CO 630, and octylphenoxypoly(ethyleneoxy) ethanol sold under the trademark IEGEPAL CA 630; and (f) from about 6 to about 12 wt% of a gelling agent.

The preferred non-caustic cleaner is manufactured by the following steps: (a) adding the builders preferably in anhydrous form to a mixer and thereafter blending the builders together; (b) adding the surfactant to the blended builders and thereafter blending the surfactant with the builders; (c) adding water after or simultaneously with the surfactants and thereafter blending the mixture until substantially all of the water forms

hydrates with the builders; (d) thereafter adding a metasilicate and/or sesquisilicate, chelate, and peroxygen compound in that order; and (e) adding the gelling agent last.

In step 24, the alkaline cleaner, particularly the non-caustic alkaline cleaner, is mixed with water. Preferably, the non-caustic cleaner is mixed with sufficient water to provide a concentration of the cleaner in water preferably ranging from about 4 to about 8 and more preferably from about 5 to about 6 g/l. The caustic cleaner is preferably mixed with water in the volumetric ratio of about 100 parts water per 1 part caustic cleaner to about 30 parts water per 1 part caustic cleaner.

The alkaline cleaner is preferably contacted with the device while the residual acid cleaner is still in contact with the device. In this manner, the basic alkaline cleaner will react exothermically with the residual acid to heat up the device and thereby thermally enhance removal of the deposit by the alkaline cleaner. In cleaning tap lines, for example, the alkaline cleaner is typically forced under pressure through the line to push residual acid cleaner out the outlet of the line. When the caustic cleaner is pushed through the line the pressurizing gas should be nonreactive with the hydroxide, such as an inert gas. Preferably, the alkaline cleaner remains in contact with the device for a time period ranging from about 5 to about 15 minutes and more preferably from about 8 to about 10 minutes.

While not wishing to be bound by any theory, it is believed that the alkaline cleaner penetrates through holes in the deposits formed by the acid cleaner and thereby is able to get beneath the remaining oxalate and proteinaceous deposits to more effectively remove them from the surface of the device.

Next, in step 28 an antimicrobial composition is contacted with the beverage dispensing device to remove any remaining microbial deposits and/or to deodorize the device. This step may be omitted if no such deposits exist after steps 20 and 24. Any suitable antimicrobial composition may be used that is capable of killing the types of microbes in the deposit. For example, in fermented beverages, the microbes include yeast, mold and various types of bacteria. Such microorganisms cause a musty, vinegar, or diacetyl odor.

A preferred antimicrobial composition is described in copending U.S. Patent Application Serial No. 09/023,347 filed February 13, 1998. The liquid composition

includes (a) from about 9 to about 11.5 wt% of a sulfonated compound (e.g., a sulfonated oleic acid, sulfonated linoleic acid, a sulfonated myristic acid, and mixtures thereof); (b) from about 4.5 to about 5.5 wt% of a solubilizing agent (a propylene glycol, ethyl alcohol, isopropyl alcohol, hexylene glycol, and mixtures thereof); and (c) from about 25 to about 35 wt% of an antimicrobial agent (e.g., phosphoric acid, citric acid, and lactic acid and esters and salts thereof). The remainder of the composition is a suitable solvent such as water, hydrogen peroxide, propylene glycol, isopropyl alcohol, and mixtures thereof.

The antimicrobial composition is made by adding the above-noted components to the solvent in any sequence. Preferably, the sulfonated compound and the solubilizing agent are added within about 5 minutes of one another.

The antimicrobial composition is typically diluted before contact with the beverage device. Preferably, the antimicrobial composition is contacted with additional solvent (e.g., water) in the volumetric ratio of from about 400 parts solvent to 1 part antimicrobial composition to about 200 parts solvent to 1 part antimicrobial composition.

The antimicrobial composition is contacted with the device commonly when residual alkaline cleaner is still on the device. For example, in a tap line the antimicrobial composition is introduced under pressure to the tap line to force the alkaline cleaner out the outlet to the line.

The residence time of the antimicrobial composition in contact with the device typically ranges from about 2 to about 10 minutes and more typically from about 4 to about 6 minutes. Where prominent odors exist in the device, the residence time is preferably lengthened to at least about 30 minutes and in the case of especially severe odor problems to as much as 24 hours or longer.

In step 34, potable water is contacted with the device to remove substantially any residual acid cleaner, alkaline cleaner, and/or antimicrobial composition. This is especially important where the device is to be used for further beverage dispensing.

The above-noted steps are repeated 38 as necessary to remove the desired amount of deposits from the device.

In another optional step 42, a second antimicrobial composition is contacted with external surfaces of the device to remove mold. The second antimicrobial composition preferably includes from about 8 to about 12 wt% of an ammonium compound such as a

quaternary ammonium compound and from about 1 to about 3 wt% of a surfactant such as nonphenol. The remainder of the antimicrobial composition is a solvent such as water. The composition is noncorrosive to stainless steel and galvanized metals. Preferably, the composition is sprayed onto the device surface where mold is present.

5           The cleaned and/or sanitized beverage dispensing device 46 is preferably substantially free of oxalate, protein and microbial deposits.

## EXPERIMENTAL

10           Various cleaning tests were performed to compare the performance of cleaning compositions of the present invention with a conventional caustic cleaner. The conventional caustic cleaner is sold under the trade name "PENETRATE". The cleaner is a potassium hydroxide product containing approximately 35 wt% hydroxide with the remainder being water. The cleaner has been the standard in the industry since the early 1980's.

15

### Experiment 1

15           In a first experiment, a cask conditioned ale line had developed a large yeast growth throughout the line. The noncaustic alkaline cleaner described above was used at a concentration of about 2.5 oz of the cleaner in 2.5 gallons of water. The aqueous  
20           noncaustic cleaner was passed under pressure through the ale line. To pass the cleaner through the line, the cleaner was pressurized with a gas containing 70% by volume nitrogen and 30% by volume carbon dioxide and held in the line for 10 minutes. After removal from the ale line, it was observed that the cleaner removed significant amounts of the yeast but significantly less than all of the yeast. The "PENETRATE" only removed  
25           small amounts in a similar line.

### Experiment 2

          A second experiment was performed using another cask conditioned ale line of similar age and condition to that in Experiment 1. The cleaning procedure was as follows:

30           (a) One ounce of the preferred acid cleaner noted above was added to 2.5 gallons of water. The solution was pushed through the ale line with a gas containing 70% by

volume nitrogen and 30% by volume carbon monoxide and allowed to stay in contact with the ale line for about 10 minutes.

(b) The alkaline cleaner prepared as described above in Experiment 1 was then pushed through line under pressure in the manner set forth in Experiment 1.

5           The procedure removed more of the yeast than in Experiment 1 but the ale line was still less than 100% clean.

### Experiment 3

10           Using another ale line of the condition of the ale line described in Experiment 1 above, the following procedure was followed:

15           (a) One ounce of an acid cleaner (i.e., 35 wt% nitric acid, 8 wt% phosphoric acid, and 3 wt% surfactant) was added to 1 gallon of water. This solution was pushed through the line using a pressurized gas including 70% by volume nitrogen and 30% by volume carbon dioxide. The acid cleaner was allowed to remain in contact with the line for approximately 10 minutes.

          (b) The noncaustic alkaline cleaner described above in Experiment 1 was next pushed through the line using the gas of step (a) in the manner described above in Experiment 1. The cleaner remained in contact with the line for approximately 10 minutes.

20           (c) An antimicrobial composition having the same composition as the preferred antimicrobial composition described above was pushed through the line using the gas of step (a) and left in contact with the line for approximately 10 minutes.

          The steps removed at least 95% of the yeast and produced a noticeable lightening of the line.

25

### Experiment 4

          After a two-week period, the line of Experiment 3 was recleaned following the procedure outlined in Experiment 3. The recleaned line had approximately 100% of the yeast removed from the line.

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### Experiment 5

A 10-year old line that had been cleaned with "PENETRATE" for those 10 years by sponging was used in the next experiment. Sponging uses a sponge saturated with "PENETRATE" that just fits in the beer line and is then pushed through the line with a pump. Sponging is first cleaning the line with "PENETRATE" then pumping a sponge through the line (it is actually forced through the line with pressure). The sponge is supposed to remove all of the deposits in the line, leaving it clean. Using the procedure as outlined in Experiment 3, the effluent of the various cleaning or sanitizing compositions contained considerable "floaty" material and a very strong vinegar odor. A vinegar odor is a direct result of bacteria removal.

This line was observed every day for two weeks. There was no return of a bitter beer taste that always returned within 3 days after a "PENETRATE" sponge cleaning.

#### Experiment 6

The line condition described in Experiment 5, that is a bitter beer flavor or off-odor, was observed in ten other lines of various lengths. The youngest line was 5 years old, and the oldest line was 12 years old. Five lines were cleaned using a carbon dioxide pot following the procedure of Experiment 3. The other five lines were cleaned using a product transfer pump following the procedure in Experiment 3. In each case, the end result was the same -- a clean line that produced clean, crisp beer for two weeks when the next cleaning was scheduled.

#### Experiment 7

A line that had a flavored beer (raspberry wheat) was cleaned using the procedure of Experiment 3. The first cleaning produced a clean line for 3 days. The second cleaning produced a clean line for 2 weeks. This line had been cleaned with straight bleach and "PENETRATE" on several occasions with the odor and flavor returning within 1 day.

The reasons for the lack of success of "PENETRATE" are believed to be at least the following:

(a) When any sodium or potassium hydroxide product is used in the presence of a carbon dioxide gas, there is a degree of neutralization due to carbon dioxide reacting

with water producing carbonic acid. This reaction reduces the alkaline present for protein peptization.

(b) No caustic cleaner on the market including "PENETRATE" currently has chelators or surfactants specific to remove oxalate scale.

5 (c) The use of pumps produces adequate velocity for cleaning; however, conventional caustic cleaners such as "PENETRATE" as mentioned in Experiment 2 do not penetrate the oxalate scale.

Reasons for the success of the methodology and cleaning compositions of the present invention include:

10 (a) The acid cleaners on the market are specific for oxalate; however, they are either phosphoric or hydrochloric acid-based and do not attack proteinaceous deposits in the same manner or to the same degree as nitric acid.

(b) No one has used the combination of using an acid cleaner first to attack the oxalate scale and also "soften" the protein deposits before using an alkaline cleaner.

15 (c) The noncaustic alkaline cleaner works not only in a carbon dioxide environment but also has a surfactant to penetrate soils and chelators for oxalate scale. This combination makes the cleaner ideal for use in both pots and pumps of beverage dispensing lines.

#### 20 Experiment 8

This trial was run on side by side lines approximately 100 feet long both having light beer in them and both being about 6 years old.

25 The first line was cleaned with a conventional caustic cleaner at a rate of 3 ounces per 2.5 gallons of cold water and allowed to stand in the lines for 10 minutes. The cleaning solution was observed to have a small amount of particles floating in the solution. The tap was then flushed with water. The next step was to prepare one packet of noncaustic alkali cleaner (50 grams) in 2.5 gallons of cold water and allow the mixture to stand in the tap line for another 10 minutes. The cleaning solution was observed after the 10 minutes to have a large amount of precipitate. The precipitate was on the order of 10-  
30 fold more than that in the effluent removed by the initial conventional caustic cleaner.



The second line was then cleaned using the noncaustic alkali cleaner followed by the conventional caustic cleaner. Both cleaners were run at the same concentration, times, and temperatures. The result was different; the noncaustic alkali cleaner removed the same amount of dirt (precipitate) as in the first line; however, the conventional caustic cleaner did not remove anything. This result indicated that the noncaustic alkali cleaner was a far superior cleaner compared to conventional caustic cleaners.

#### Experiment 9

The following field trials were run over a period of two months from January 4 through March 8, 1999. The trials were run on a number of existing beer dispensing lines. The types of beer, condition, and styles of the lines were kept to a constant as much as possible.

The following trials were run using the acid cleaner, noncaustic alkali cleaner and antimicrobial composition. The lines tested were high density polyethylene, their ages were approximately 5 years, and their lengths were up to 150 feet.

Each solution was prepared as follows;

- One packet of noncaustic alkali cleaner in 2.5 gallons of cold water.
- Two ounces of acid cleaner in 2.5 gallons of cold water.
- One ounce of antimicrobial composition in 2.5 gallons of cold water.
- Each mixture was placed sequentially and discretely in a 2.5 gallon CO<sub>2</sub> pot.
- Each mixture remained in the line for about 10 minutes.

The acid cleaner was run first. The cleaner yielded different results on two different lines. When the acid cleaner was removed from the first line, the cleaner had a very dark color similar to that of actual beer, but there was no visible residue produced. In this line it was noted that oxalate scale was not present. When the acid cleaner was removed from the second line, the cleaner had a white heavy precipitate. In this line it was noted that a heavy oxalate scale was present. The final cleaning solution had the appearance of buttermilk or thin yogurt.

A noncaustic alkali cleaner was run next, directly after the acid cleaner in both trials. A very heavy precipitate was contained in the noncaustic alkali cleaner upon

removal. It was noted that at no time had this amount of dirt ever been removed from either of the two lines without the aid of some type of scraping device. One of the lines produced a soil that resembled tobacco flakes. The flakes were so heavy that they filled the entire solution, producing a dark color when agitated. At rest, the soil settled out of the cleaner, filling one-half of a glass containing the removed cleaner. The second line produced another heavy white precipitate that actually resembled the soil that was removed when the acid cleaner was used.

The antimicrobial composition was run last. Upon removal it did not have any color or precipitate. It was run through the lines to "freshen" the line rather than clean it.

The final lines were examined and found to be noticeably clean. In the case of the first line, it was noted that the very heavy brown color was gone. This would account for the color noted in the first line experiment. In the case of the second line it was noted that the white build up of oxalate in the line was about 95% removed. The beer coming out of these two lines after this experiment was noticeably fresher tasting. After two weeks the odors noted before this trial still had not returned.

#### Experiment 10

Following the same procedures in Experiment 9 but changing the protocol by running the noncaustic alkali cleaner first; the acid cleaner second; and the antimicrobial composition last. The results are as follows:

1. The noncaustic alkali cleaner removed the volume of precipitate noted in Experiment 8.

2. The acid cleaner removed a good portion of oxalate soil; however, when inspecting the line there was a visible amount of organic material left in the line. It is theorized that this organic material was deposited under the oxalate.

3. The antimicrobial composition failed to remove any additional organic material; however, the strong stale beer odor was noticeably removed.

4. The line was about 85 to 90% cleaned.

#### Experiment 11

Following the same procedures as outlined in Experiment 9 but changing the order of use to antimicrobial composition first, noncaustic alkali cleaner second, and acid cleaner third, the following results were observed:

1. The antimicrobial composition had no visible cleaning whatsoever.
2. The noncaustic alkali cleaner removed about the same or perhaps a little more deposited than it normally would. The soil load in the line had a tobacco-like appearance rather than the normal flake appearance. This could be due to the line rather than the order of addition.
3. The acid cleaner removed oxalate scale; however, the net result was the same as in Experiment 10 with only a small amount of protein soil remaining on the line.
4. The cleaning efficiency was similar to that of Experiment 11.

#### Experiment 12

In this experiment the order of cleaning was acid cleaner first, antimicrobial composition second, and noncaustic alkali cleaner last. The end results were as follows:

1. The acid cleaner turned white as described in the second line cleaning in Experiment 9. The precipitate was very heavy indicating a large oxalate build up.
2. The antimicrobial composition had a small amount of particles in it, probably the result of flushing rather than the result of a cleaning action.
3. The noncaustic cleaner removed an equal amount of precipitate. Oddly the removed noncaustic alkali cleaner had basically the same color as the oxalate removed by the acid cleaner.

The net result was a line that was about 90 to 95% clean; however, the sanitizing step was in the wrong order which after 3 days became evident. The line in this trial began to show a significant amount of microorganism growth after three days whereas the line in Experiment 9 did not demonstrate growth until the 12th day.

In summary, the order of addition is important when treating lines that are scaled with oxalate, have off odors in them, are contaminated with microorganisms, or have been neglected. When any of these conditions are present, the order of addition listed in Experiment 9 appears to be important. It appears to be the only order that will produce consistent cleaning, resulting in a clean, uninfected, and odor-free line.

The use of only the noncaustic alkali cleaner as a one-step cleaner appears to be insufficient. Lines that had been cleaned on a 2-week regimen eventually developed oxalate deposits and required the cleaning procedure outlined in Experiment 9 for thorough removal of deposits.

5           The use of acid cleaner as a one-step cleaner is also insufficient for cleaning. By itself the acid cleaner cleans lightly soiled lines; however after repeated use protein soils can build up in the lines and cause odor problems.

          The antimicrobial composition is an excellent sanitizer and odor remover; however it will do nothing to remove oxalate and protein deposits. This fact indicates that it should  
10           only be used as a last step product once the line is at least 90% clean.

          The development of the acid cleaner was the last piece to the cleaning puzzle. After the cleaner's development, lines that had been written off or scheduled for removal were not only cleaned but rejuvenated back to reuse.

          The foregoing description of the present invention has been presented for purposes  
15           of illustration and description. Furthermore, the description is not intended to limit the invention to the form disclosed herein. Consequently, variations and modifications commensurate with the above teachings, in the skill or knowledge of the relevant art, are within the scope of the present invention. The embodiments described here and above are further intended to explain best modes for practicing the invention and to enable others  
20           skilled in the art to utilize the invention in such, or other, embodiments and with various modifications required by the particular applications or uses of the present invention. It is intended that the appended claims be construed to include alternative embodiments to the extent permitted by the prior art.

What is claimed is:

1. A method for cleaning and sanitizing food processing equipment and beverage dispensing devices, comprising:

(a) contacting an article having at least two of a mineral deposit, a protein deposit, and a microbial deposit with an acidic cleaning composition to at least partially remove any mineral deposits;

(b) contacting the article with an alkaline cleaning composition to at least partially remove any protein deposits; and

(c) contacting the article with an antimicrobial composition to at least partially remove any microbial deposits.

2. The method of Claim 1, wherein the acidic cleaning composition includes at least one of nitric acid, sulfuric acid, hydrochloric acid, and mixtures thereof.

3. The method of Claim 1, wherein the acidic cleaning composition includes nitric acid and phosphoric acid.

4. The method of Claim 3, wherein the acidic cleaning composition includes a surfactant.

5. The method of Claim 1, wherein the alkaline cleaning composition includes at least one of a hydroxide, a peroxygen compound, and mixtures thereof.

6. The method of Claim 1, wherein the alkaline cleaning composition includes:

from about 25 to about 35 wt% of a peroxygen compound;

from about 25 to about 35 wt% of a silicate; and

from about 2 to about 8 wt% of a chelate.

7. The method of Claim 1, wherein the alkaline cleaning composition includes:

from about 35 to about 40 wt% of a hydroxide; and

from about 2 to about 3 wt% of a gluconate.

8. The method of Claim 1, wherein the alkaline cleaning composition has a pH of about pH 7 or greater.

9. The method of Claim 1, wherein the antimicrobial composition includes phosphoric acid and an anionic detergent.

10. An article treated by the method of Claim 1.

11. A process for cleaning and sanitizing beverage dispensing devices, comprising:

(a) contacting a beverage dispensing device having at least one of a mineral deposit, a protein deposit, and a microbial deposit with an acidic cleaning composition to at least partially remove any mineral deposits and

(b) contacting the device with an alkaline cleaning composition to at least partially remove any protein deposits.

12. The process of Claim 11, wherein the acidic cleaning composition includes:

from about 15 to about 25 wt% nitric acid;

from about 8 to about 15 wt% phosphoric acid; and

from about 1 to about 4 wt% surfactant.

13. The process of Claim 11, wherein the alkaline cleaning composition includes:

from about 25 to about 35 wt% of a peroxygen compound;

from about 25 to about 35 wt% of a silicate; and

from about 2 to about 8 wt% of a chelate.

14. The process of Claim 11, wherein the alkaline cleaning composition includes:

from about 35 to about 40 wt% of a hydroxide; and

from about 2 to about 3 wt% of a gluconate.

15. The process of Claim 11, further comprising:

(a) contacting the device with a antimicrobial composition to at least partially remove any microbial deposits.

16. The process of Claim 15, wherein the antimicrobial composition includes phosphoric acid and anionic detergent.

17. A beverage dispensing device treated by the process of Claim 10.

18. An acidic cleaning composition, comprising:

(a) an aggressive acid that is at least one of nitric acid, sulfuric acid and hydrochloric acid; and

(b) a stabilizing acid that is at least one of phosphoric acid, oxalic acid, citric acid, and hydroxyacetic acid to stabilize the aggressive acid.

19. The acidic cleaning composition of Claim 18, wherein the aggressive acid is nitric acid and the stabilizing acid is phosphoric acid.

20. The acidic cleaning composition of Claim 18, further comprising a surfactant.

21. The acidic cleaning composition of Claim 18, wherein the pH of the composition is less than about pH4.

22. The acidic cleaning composition of Claim 18, wherein the concentration of the aggressive acid ranges from about 2.5 to about 6 mole/liter.

23. The acidic cleaning composition of Claim 18, wherein the concentration of the stabilizing acid ranges from about 1 to about 3 mole/liter.

24. The acidic cleaning composition of Claim 18, wherein the molar ratios between the aggressive acid and the stabilizing acid ranges from about 2:1 to about 3:1.

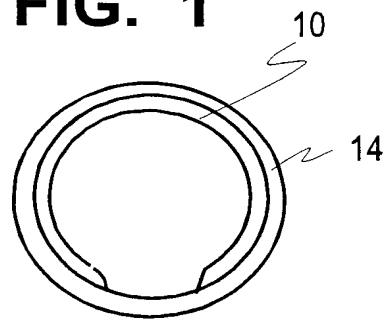
25. The acidic cleaning composition of Claim 20, wherein the surfactant is selected from the group consisting of nonophenols, octylphenols, polyoxyethylene-polyoxypropylene glycols, phosphate esters, ethylene oxides, and mixtures thereof.

26. The acidic cleaning composition of Claim 20, wherein the concentration of the surfactant ranges from about 0.006 to about 0.03 moles/liter.

27. The acidic cleaning composition of Claim 20, wherein the molar ratio between the aggressive acid and the surfactant ranges from about 350:1 to about 100:1.

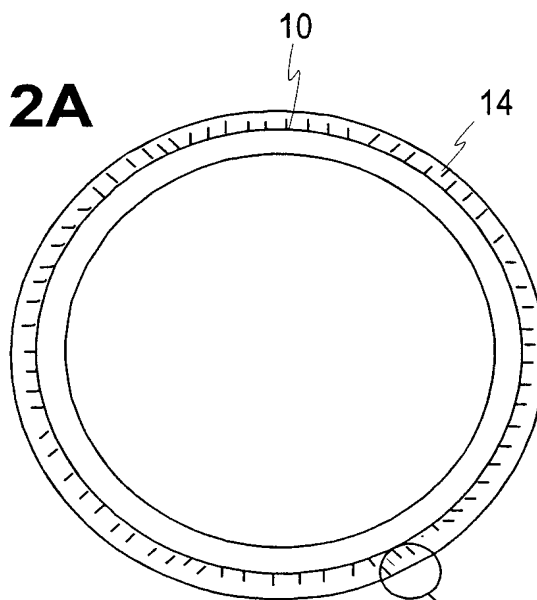
1/2

**FIG. 1**



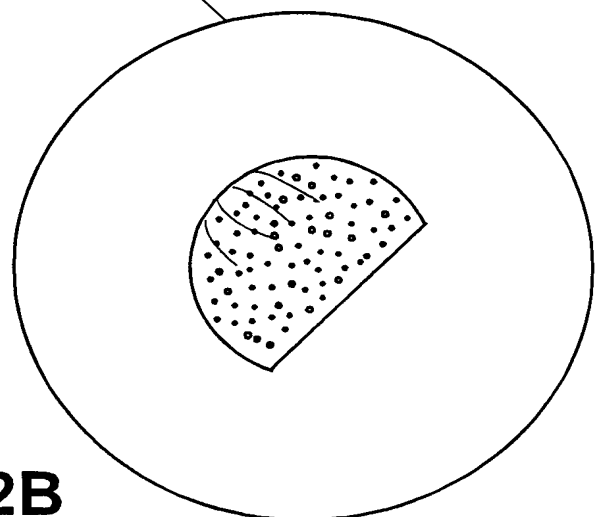
PRIOR ART

**FIG. 2A**



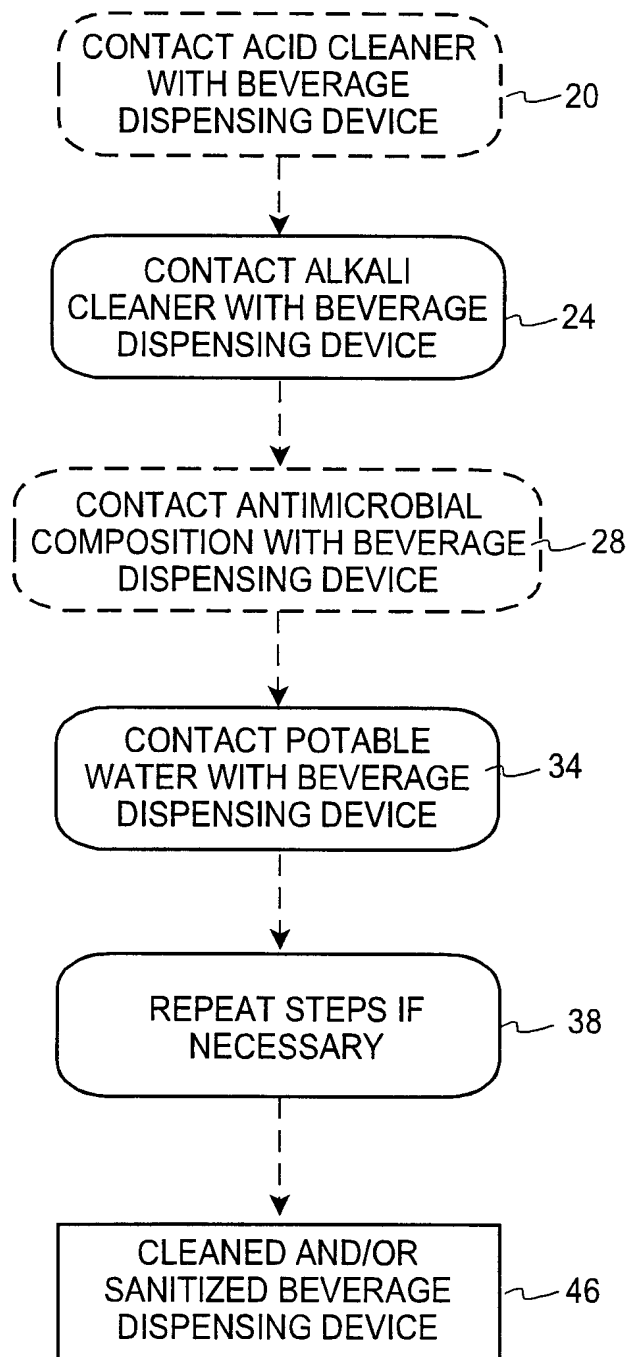
PRIOR ART

**FIG. 2B**



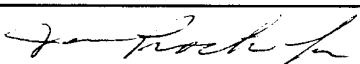


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**FIG. 3**

## INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US00/10177

<b>A. CLASSIFICATION OF SUBJECT MATTER</b> IPC(7) :C11D 7/08, 3/08, 1/72, 7/06 US CL :Please See Extra Sheet. According to International Patent Classification (IPC) or to both national classification and IPC		
<b>B. FIELDS SEARCHED</b> Minimum documentation searched (classification system followed by classification symbols) U.S. : 510/218, 207, 247, 252, 253, 254, 269, 272, 309, 363, 367, 372, 378, 511, 512, 534 Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) WEST 2.0 SEARCH TERMS: NITRIC, SULFURIC, HYDROCHLORIC		
<b>C. DOCUMENTS CONSIDERED TO BE RELEVANT</b>		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4,212,761 A (CIACCIO) 15 July 1980, col. 5, example 1.	1-27
Y, P	US 5,898,024 A (TALLEY) 27 April 1999, col. 12, example 7.	1-27
Y, P	US 5,998,358 A (HERDT et al) 07 December 1999, col. 14, example 2.	1-27
Y	US 5,424,000 A (WINICOV et al) 13 June 1995, col. 5, example 3.	1-27
<input type="checkbox"/> Further documents are listed in the continuation of Box C. <input type="checkbox"/> See patent family annex.		
* "A" "E" "L" "O" "P"	Special categories of cited documents: document defining the general state of the art which is not considered to be of particular relevance earlier document published on or after the international filing date document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) document referring to an oral disclosure, use, exhibition or other means document published prior to the international filing date but later than the priority date claimed	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art "A" document member of the same patent family
Date of the actual completion of the international search 03 JUNE 2000		Date of mailing of the international search report 07 JUL 2000
Name and mailing address of the ISA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703) 305-3230		Authorized officer  CHARLES BOYER Telephone No. (703) 308-0661

# INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US00/10177

## A. CLASSIFICATION OF SUBJECT MATTER:

US CL :

510/218, 207, 247, 252, 253, 254, 269, 272, 309, 363, 367, 372, 378, 511, 512, 534